Mini Review

Novel components in anion exchange membrane water electrolyzers (AEMWE's): Status, challenges and future needs. A mini review

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ABSTRACT

AEMWE is a novel technology combining the advantages of the already recognized electrolyzer technologies, i.e. Proton Exchange Membrane WE (PEMWE) and Alkaline WE (AWE) with the promise to eliminate the disadvantages of both. This review presents an overall opinion of the current state of AEMWE technology, focusing on component's performance, durability and overall operation while identifying the critical gaps in the technology. It presents our perspective on the requisite developments in AEMWE at the cell component level in order to become a viable technology amongst other electrolyzer technologies with the potential for widespread adoption and commercialization.

1. Introduction

Prospects of operation using inherently cost-effective components such as stainless steel bipolar plate (BPP)/Flow Field (FF)/porous transport layer (PTL) and non-platinum group metal (non-PGM) catalysts separated with AEM with gas pressurization capability and fast response to intermittent loads from renewables, makes AEMWE's an attractive technology for producing clean and low cost hydrogen. Estimations report up to 20% investments costs for AEM compared to PEMWE and H_2 production going down to 1.5–3 € kg^{-1} within the next 5 years (see SI).

For commercialization and widespread adoption, AEMWE's performance needs to match or exceed especially PEMWE's high purity of hydrogen, high operating current density (CD) and system performance, lifetime, and pure water operation[1]. This review presents a relevant selection of recent developments in AEMWE to identify the critical gaps in technology and further motivate the requisite advancements in the field as provided by our perspective.

2. Catalysts: Hydrogen evolution reaction (HER), oxygen evolution reaction (OER)

HER kinetics of non-PGM catalysts in AEMWE's alkaline setting are generally 2–3 orders of magnitude slower than in acidic PEMWE[2]. In alkaline solutions HER entails a water discharge step, the Volmer step

($H_2O + e^- \leftrightarrow H_{ad} + OH^-$) followed by either the electrochemical Heyrovsky step ($H_2O + H_{ad} + e^- \leftrightarrow H_2 + OH^-$) or the chemical Tafel recombination step ($2H_{ad} \leftrightarrow H_2$) [2] corresponding to Tafel slopes of 120, 40 and 30 mV/decade, respectively[3] with the largest slope indicating the rate determining step. In acid solutions, H_3O^+ is the proton donor and thus energetics of H_{ad} is a major activity descriptor. With water as opposed to H_3O^+ as proton donor in alkaline environments, however, a bifunctional catalyst that can stabilize both parts of water i.e. H^+ and OH^- to form an activated water complex leads to enhanced HER rate. Water cleavage "promoter" oxides/hydroxides has been utilized to remarkably enhance catalytic activity of electrocatalysts in water splitting[4]. In particular, $Ni(OH)_2$ is shown to help stabilize OH^- whose addition to otherwise not so active catalysts results in HER rate enhancement[4]. $Ni(OH)_2$ electrodeposited on tungsten nitride nanowires (WN-$Ni(OH)_2$) exhibits Tafel slope of 96 mV/dec in contrast to 118 and 110 mV/dec for its constituents WN and $Ni(OH)_2$. This attests to the synergistic interaction of WN and $Ni(OH)_2$ by providing active sites for H_{ad} during water cleavage and facilitating OH_{ad} desorption leading to enhanced HER activity.

A recent study[5] reports a bifunctional system of NiCu mixed metal oxides (MMO) consisting of Ni, NiO and CuO and in-situ generated hydroxide species $Ni(OH)_2$ and $Cu(OH)_2$, whose presence and retention were proven with in-situ Raman spectroscopy, providing superior HER through enhancement of Volmer step kinetics. This improvement is attributed to NiO which facilitates water dissociation and stabilizes

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OH_{ad} while Ni metal and CuO stabilize H_{ads} and NiO, respectively. In contrast, NiCu alloy and NiCu oxide indicated inferior activity compared to NiCu MMO.

Chen et al.[6] annealed a precursor of $NiMoO_4$ on Ni foam in NH_3/H_2 (5% H_2, 550 °C) obtaining a $NiMo-NH_3/H_2$ catalyst that required an overpotential of only −11 mV to achieve −10 $mA/cm[2]$ with a Tafel slope of 35 mV/dec. The same catalyst was subjected to anodic oxidation, 1.3 → 1.5 V *vs.* RHE (Reversible Hydrogen Electrode) in 1 M KOH in the presence of Iron ions to obtain $Fe-NiMo-NH_3/H_2$ that also exhibited substantial OER activity. A Membrane Electrode Assembly (MEA) was prepared consisting of $NiMo-NH_3/H_2$ and $Fe-NiMo-NH_3/H_2$ deposited on carbon paper as anode and cathode, respectively, separated by a X37-50 Sustainion AEM (50 μm, Dioxide Materials). AEMWE cell equipped with this MEA indicated an impressive operation of 1 A/cm^2 @ 1.57 V, 80 °C with 1 M KOH.

Xu et al. [7] reported complex oxides of Mo with scheelite $BaMoO_4$ taken through a thermal-reduction route to obtain $BaMoO_3$ perovskite. $BaMoO_3$ compared to $BaMoO_4$ has the following features all favoring Volmer step: (1) A higher symmetry and collinear covalent bridge connection of active sites -Mo-O-Mo-O- offers a shorter reaction path (2) Mo in $BaMoO_3$ has a lower oxidation state compared to $BaMoO_4$ (+4 *vs.* + 6) leading to increased d-band filling of Mo and thus better tuning of the H_{ad} energy in the Volmer step, (3) presence of oxygen vacancies on $BaMoO_3$ can facilitate water dissociation in the Volmer step (4) the electrical conductivity of $BaMoO_3$, 1.4×10^2 S/cm, is >7 orders of magnitude larger than $BaMoO_4$. The result is a Tafel slope of 110 mV/dec with superior HER performance compared to perovskites of other transition metals such as Mn, Fe, Co and Ni and the widely studied Mo-based catalysts, MoS_2.

OER involves consuming the OH^- anions generated at the cathode via cleavage of water which are transported through the AEM: $4OH^- \leftrightarrow 2H_2O + O_2 + 4e^-$. Sluggish by nature, the state-of-the-art OER catalyst is the PGM-based IrO_x. However, non-PGM OER catalysts with performances comparable or better than IrO_x have already been fabricated and reviewed extensively (e.g.[8]) and will be referred to only in support of other topics.

There is growing emphasis on self-supported catalysts and free-standing films directly on the electrode substrate, which omit the need for binders or additives[9,10]. More work is needed to evaluate the durability of these structures at high current densities and under practical operating conditions. Scalability may also be an issue, as the preparation of these catalyst structures on electrode supports commonly require high temperatures[11], resulting in greater energy utilization.

3. Membranes & ionomers

While it is important to characterize AEM's properties such as hydrogen diffusivity, ionic conductivity, electrolyte permeability, swelling ratio,[12] thorough AEM stability studies are lacking. Generally, AEM's are made of a polymer backbone with covalently attached cationic side chains to facilitate the movement of anions i.e. OH^-. Common failure modes of AEMs include mechanical breakage, thinning, pin-hole formation[12], poor chemical stability in high pH environments[13], absorption of CO_2[14,15] and in the long-run, loss in number of anion exchange groups either by Hoffman elimination or nucleophilic attack by OH^-[16–18]. These possible failure modes can result in short circuits, gas crossover and safety concerns[19,20] decrease in OH^- conductivity[17] and water mobility[15].

Mechanical stability involves structural composites and cross-linking, whereas chemical stability is dependent on harnessing suitable side chains[18]. The proximity of side chains to the polymer backbone affects chemical stability and spacers are suggested to assist in optimizing this distance[14]. Promoting the mobility of OH^- through the membrane is tantamount to enhancing the ion exchange capacity (IEC: number of exchangeable ions/membrane dry weight, mmol/g). However, this also contributes to a greater water uptake and swelling

[21] that in turn reduces the mechanical stability. Several groups have tackled this issue by blending one polymer for mechanical strength with another to assist with ionic conductivity[22–24]. Aili et al.[25] fabricated homogenous membranes from polymer blends of poly(arylene ether sulfone) or PSU and poly(vinylpyrrolidone) or PVP and found out that blends with > 45% PVP showed ionic conductivities of 0.01–0.1 S/cm whilst retaining mechanical strength. AEMWE cell level stability test conducted over a week at 500 mA/cm^2 in 20 wt% KOH at 60 °C with the best performance obtained at 1.9–2 V and a long term test > 700 h resulted in voltages 2–2.1 V. Similarly, a polymer blend of 35 wt% poly (ether ketone-cardo) (PEK-cardo) and poly(vinyl benzyl chloride) PVBC functionalized with 1-methylpyrrolidine cationic groups showed a good ionic conductivity of up to 0.03 S/cm and mechanical strength of 15.1 MPa and reported lifetime cell testing performance using non-PGM catalysts at 500 mA/cm^2 in 1 M KOH, 60 °C with cell voltage 2.0–2.1 V and degradation rate of 3 mV/h over 46 h [26].

A second strategy constitutes blending polymers with ionomers: Henkensmeir et al.[27] showed that adding anion exchange ionomer (AEI), FAA3 to polybenzimidazole (PBI) improved the tensile strength, up to 93 MPa in 25 wt% KOH, and raised the ionic conductivity of the membrane to a maximum of 0.166 S/cm in 25 wt% KOH which is comparable to Nafion117 in 1 M H_2SO_4[28]. However, AEM aging tests indicated the degradation of both properties although to a lesser extent than in un-doped PBI. Stability tests in AEMWE cells equipped with these membranes maintained 2 V over 100 h at 200 mA/cm^2 in 20 wt% KOH, 60 °C. Alternatively, ion-solvating membranes which can be described as a polymer matrix doped with the supporting electrolyte of the system such as KOH or NaOH have been proposed thus eliminating ionomer/side chain susceptibility to degradation by the OH^- ions[29]. Further progress is still required to find chemically stable ion-solvating polymers with high ionic conductivity[30] on par or better than the commercial membranes (>0.166 S/cm).

The performance of commercial Aemion™, AF1-HNN8 AEM's developed from hexamethyl-p-terphenyl poly(benzimidazolium) was determined in combination with ionomers supplied by Ionomr Innovations Inc. AP1-HNN8 or FumaTech's Fumion FAA-3 AEI. Fuma-Tech's FAA-3–50 AEM based on a brominated polysulfone backbone with side chain groups of quaternary ammonium was assembled in a cell with $NiMnO_x$ and Pt/C as OER and HER catalysts, respectively and operated in 1 M KOH, 50 °C pumped to the anode side[31]. The stability was assessed through voltage cycling 1 ↔ 1.8 V over 1000 h resulting in 5% loss in voltage efficiency. Typically in PEMWE, hydrogen is pressurized to up to 30 bars or more. AEMWE operation with H_2 pressurization up to 8.5 bars is reported by Ito et al.[32] using ultra-thin AEM (A201, 28 μm, Tokuyama) with H_2 content in O_2 (crossover) of only 0.16 of that in a PEM, which was associated with AEM's H_2 barrier capability. Notably. the cathode electrode contained Pt/C catalyst and AEI (AS-4, Tokuyama) while anode electrode was based on $CuCoO_x$ catalyst with PTFE binder.

4. MEA fabrication and long-term performance evaluation

MEA is at the heart of AEMWE and its structure plays an important role in performance and durability. Primarily two different MEA fabrication methods are proposed: catalyst-coated substrate (CCS) and catalyst-coated membrane (CCM)[33,34], each with pros and cons. In the CCS method, catalyst ink, consisting of a binder/ionomer, catalyst nanoparticles and solvents, is applied to one side of the PTL to form the catalyst layer (CL) whereas in CCM method the catalyst ink is applied to the AEM to form the CL. Small cells were assembled with a A201, 28 μm AEM, Tokuyama with IEC of 1.7 mmol/g along with commercial catalysts Acta-4030 (Ni nanostructure/CeO_2-La_2O_3/C) and Acta 3030 ($CuCoO_x$) as cathode and anode catalyst materials, respectively and tested to evaluate the effect of MEA fabrication methods[33]. CCM method is generally considered superior to CCS because it ensures better contact with the membrane and thus efficient mass transport of ions.

However, AEI AS-4, (Tokuyama) indicated low mechanical/chemical stability for anode CL. Therefore, the anode CL was fabricated with the CCS method with PTFE as a binder. Since there was no electrolyte flow through the cathode, the CCM method on the cathode side, yielded stable results. The optimal configuration of CCS-anode/MEM/CCM-cathode was therefore obtained, indicating that the AEI plays an important role in MEA forming ionic pathways in the CL and as catalyst binder.

Another study[34] investigated CCM and CCS methods using FAA-3–50 AEM and AEI/binder FAA-3-Br (Fumatech) with highly active PGM catalysts to deconvolute the investigation from catalytic activity effects. The results showed that hot-pressing of CCS onto AEM provided better ionic connection. Although this resulted in improved performance, the CCM method still yielded the highest CD's of 1.15 and 1.5 A/cm^2 at 1.8 and 1.9 V, respectively.

A similar setup with FAA-3-PK-75 AEM (Fumatech) either sandwiched or hot pressed between two CCS-fabricated electrodes with PGM catalysts[35] was used to study the effect of hot-pressing conditions. The CL's, however, contained PTFE as binder which is not ionically conductive. In this case hot pressing was deemed detrimental to the performance of AEMWE due to the PTFE binder covering the catalytic sites and causing structural deformation. Using CCS-fabricated MEAs based on non-PGM catalysts namely, NiFe$_2$O$_4$ on nickel PTL (cathode) and NiFeCo on stainless PTL (anode), with Nafion as binder and three different membranes Sustanion, A-201, and Ameion™, a high CD of 2 A/cm^2 could be achieved at 2.13, 2.26 and 2.21 V, respectively all operated with 1 M KOH @ 60 °C[36]. Post mortem MEA analysis indicated the significance of the AEI: only Sustanion had well adhered CL's while other membranes indicated weakly bonded and delaminated CL's likely due to mismatch in thermal and hydrated expansion properties.

Recently, Faid et al.[37] embedded a reference electrode into a AEMWE cell to measure and distinguish the effect of AEI contents on anode and cathode performance. Non-PGM catalysts of Ni/C (cathode) and NiO (anode) mixed with either Fumion FAA-3 (Fumatech) AEI or Nafion binder were sprayed onto Toray 090 carbon paper that in turn was used to sandwich a FAA-3-PE-30 (Fumatech) AEM to make MEA (CCS). For OER though, the NiO anode CL potential was nearly independent of AEI content within the range investigated (10–40 wt%). Using Rotating Disk Electrode (RDE) studies for HER, CL with AEI was shown to have a charge transfer resistance twice that of CL with Nafion binder indicating a lower HER activity. The difference is due to the specific adsorption of cation functional group, quaternary ammonium, present in AEI causing HER reactants to experience a lower effective potential.

A newly published work presents an AEMWE which utilizes a 2 wt% zirconia-mechanically reinforced 50 μm Sustainion® AEM (X37-50) sandwiched between NiFe$_2$O$_4$ (2 mg/cm^2) on stainless-steel porous plate and Raney Nickel (3 mg/cm^2) on nickel fiber paper as anode and cathode, respectively. The 5 cm^2 cell operated with 1 M KOH at 60 °C indicated a stable operation at 1 A/cm^2 at 1.85 V for an impressive 10,000 hrs (20 years lifetime) with degradation rate of less than 1 μV/h [38]. An accelerated stress test (AST) simulating on/off duty cycles of the renewables, was applied to the cell by cycling the CD 0.02 ↔ 2.4 A/cm^2 for 11,000 times with 2 s hold at each CD. This caused a "voltage shock" on each cycle (1.6 ↔ 2.1 V) that resulted in a performance loss of ~ 0.15 μV/cycle with electrolyte flowing at all times.

Rest time/frequency of AST cycles are shown to have significant impact on performance[39] with recoverable and irreversible losses to be differentiated. In another work, anode and cathode nanoparticles of NiFe$_2$O$_4$ and NiFeCo with Nafion as binder were spray-coated onto stainless steel fiber cloth and carbon cloth, separated with a Sustanion (X37-50) AEM, collectively forming cells that were operated with 1 M KOH at 50 °C. Two different operation modes were applied to the cells potentiostatically (1.95 V) for 1000 hrs. For the first, a 12 h open circuit voltage (OCV-zero CD) with no solution feeding was applied after each 2–10 h of operation, while for the second protocol, OCV was applied after each 100 h of operation. The results indicate increased ohmic losses due to the frequent periods of OCV led to a lower humidity level concomitant with AEM degradation while losses were mostly non-ohmic and recoverable due to bubble resistance using the second protocol. The results indicate the importance of continuous electrolyte feed even at the rest times. PTL and FF

5. Pure water AEMWE operation

The majority of studies demonstrate AEMWE operation with corrosive electrolyte, typically 1 M KOH. Pure water operation for AEMWE, just like PEMWE, means simpler balance of plant (BOP), less passivation for e.g., BPP and no shunt currents all translating into lower costs. Pure water operation, however, poses challenges to membrane and catalyst and has been studied only by a few groups. Recent work[40] proves the discrepancy between *ex-situ* and *in-situ* activity evaluations: the best OER catalyst NiFeO$_x$H$_y$, substantially better than IrO$_x$, in 3-electrode measurements with 1 M KOH was the worst in pure water anode-fed AEMWE cell operation. In contrast, NiCoO$_x$-based spinel catalysts that indicate subpar performance in *ex-situ* tests perform better than IrO$_x$ in AEMWE. This is explained by the poor electrical conductivity of NiFeO$_x$H$_y$ (6.3x10^{-9} S/cm) compared to NiCoO$_x$-based spinels (1–10 S/cm). Short term stability evaluated at 0.2 A/cm^2 for 3 hr at 50 °C concluded that all catalysts, including IrO$_x$, showed poor durability indicated by voltage increase of 150–250 mV after 3 h at 0.2A/cm^2, 50 °C hinting to a common degradation mechanism. Deterioration of the ionomer which also binds the catalyst particles together and the resulting loss of electrical connectivity of anode catalyst is stated as the most likely cause of degradation. This conclusion is supported by other studies[41]: the adsorption of phenyl group in the ionomer backbone onto the catalyst produces acidic phenol at high OER anode potentials causing performance decay. Also the formation of phenol at the catalyst-ionomer interface, reduces the local pH by neutralizing the ionomer's quaternized hydroxide functional groups leading to poor activity of the otherwise superior OER catalysts in alkaline environment. DFT calculations further indicate weaker interaction of the phenyl group with non-PGM perovskite oxides compared to IrO$_x$ which makes the latter more susceptible to degradation.

RDE studies[42] proved that both OER and HER activities significantly increased with pH (12 → 14) suggesting that an AEI with higher IEC is favoured for high performing AEMWE with the drawback of increased water solubility. Using phenyl-free aliphatic polymer backbone with an ammonium or amine group substituting the phenyl group's side chains to maintain a high pH and avoid phenol formation, ionomers with relatively high IEC's (2.2–3.3 mmol/g) were fabricated [42]. With NiMo/C and NiFe catalsyst for HER and OER, respectively, resulted in an MEA activity of 0.9 A/cm^2 at 1.8 V, 60 °C in pure water operation.

Ni-foam[43] as self-supported electrode serving as both PTL and catalyst support for OER in the form of vertically grown fluoride-incorporated NiFe oxyhydroxide nanosheet arrays was used along with Pt/C HER catalyst in pure-water anode-fed AEMWE cell producing ~ 1 A/cm^2 at 1.8 V, 90 °C. Maintaining high concentration of OH$^-$ around the catalyst by means of high IEC AEI is a critical factor in achieving high performance AEMWE. As such they used a high IEC (3.2 mmol/g) PAP-TP-85-MQN AEI supplied by W7Energy and an ultra-thin 20 μm PAP-TP-85 AEM to record a reasonable degradation rate of 0.56 mV/h during 160 h of durability test at 0.2 A/cm^2.

6. Conclusions and perspective

AEMWE technology is generally considered relatively immature with the majority of the research efforts focused on developing novel components with enhanced performance and durability along with proof of concept (TRL 2–3)[44].

Our perspective is substantiated and formulated with the ultimate aim of matching and exceeding the performance of other electrolyzer

technologies esp. PEMWE's:

- Catalysts: there is an urgent need to develop highly active and durable non-PGM catalysts for the sluggish HER and OER in alkaline environment. In particular, catalysts with less sensitivity to local pH variations are sought. Conversely, catalysts with weaker interactions with AEI around the active areas to avoid pH fluctuations are worthwhile to consider. Self-supported catalysts can be advantageous, yet their long-term stability has to be validated.
- Membranes: novel AEM's with high IEC and simultaneously high mechanical/chemical stability in alkaline environment, ideally exceeding ionic conductivity for PEM while maintaining an effective hydrogen barrier capability are needed.
- Ionomers: novel AEI's are needed with higher IEC that are devoid of moieties undergoing (electro)chemical change with adverse effect on AEI degradation, local pH and catalytic performance.
- PTL/FF: in contrast to PEMWE, e.g. [45–47], studies on the effect of PTL pore size/morphology on mass/charge transport limitations in AEMWE are lacking. These investigations are required for optimal design of PTL/FF in AEMWE.

Despite the immaturity of the AEMWE technology companies such as Enapter and Alchemr have introduced commercial products as a disruptive technology, albeit without pure water operation. As attested to in this review, AEMWE components performance are reported at sporadically chosen conditions and thus standardization is required to systematically measure progress.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi. org/10.1016/j.elecom.2021.107140.

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